Study of PATP Impact on Food Packaging Materials

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The impact of pressure-assisted thermal processing (PATP) on selected polymeric food packaging materials was studied. Two commercially available packaging materials called APP (combination of biaxially oriented and PVdC coated polyester with a sealing layer against polypropylene) and BPE (combination of biaxially oriented polyester with coextruded barrier film of the structure polyethylene/EVOH/m-polyethylene) were pressurized at 600 MPa for 10 min, at 70°C. The impact of combined pressure-heat treatment on the change of packaging materials was assessed by SEM (Scanning Electron Microscopy). In addition, visual examination was performed for the treated APP and BPE materials. The results showed that the combined extreme pressure-heat treatments can compromise the integrity of both packaging materials. Opaque areas, delaminations and dark spots were a general consequence of the pressure-assisted thermal processing of tested packaging materials.

Keywords: food packaging materials; pressure-assisted thermal processing; opaque areas; delaminations

Food packaging is an essential part of food processing operations [1]. To increase the effectiveness of food processing, the packaging materials must be carefully selected [2] given their direct impact on food stability and shelf life. The choice of the packaging materials for high pressure processing should guarantee that the high pressure and severe stress regime associated with the combined thermal and pressurization-depressurization process do neither affect package integrity nor its functional properties [35]. Moreover, these materials have to present enough flexibility to compensate for the collapse of the head space and for the possible volume reduction of the food inside the package [2, 4-9]. Extra performances must be demonstrated by the packaging materials if high pressure is combined with high temperatures [5]. In this context, a really relevant issue is that packaging materials can, in some cases, suffer delamination phenomena leading to unacceptable changing of the aspect as well as losses of the integrity of the packaging structure [5]. Furthermore, the aesthetic qualities of the packaging materials should not be compromised [4, 7].

Very limited information is available on the impact of combined pressure-heat treatment on packaging material during PATP (pressure assisted thermal processing) treatments [4-5, 9-11].

The objective of this research was to investigate the impact of PATP (pressure-assisted thermal processing) at 600 MPa combined with 70°C for 10 min on *APP* (combination of biaxially oriented and PVdC coated polyester with a sealing layer against polypropylene) and *BPE* (combination of biaxially oriented polyester with coextruded barrier film of the structure polyethylene/EVOH/m-polyethylene) - packaging materials commercially available for the high pressure treatment.

This study is contributing to the general understanding of the impact of combined pressure-heat treatment on packaging materials. Moreover, the packaging materials influence on the safety and the shelf life properties of the foodstuffs was considered.

Experimental part

Materials and methods

Packaging materials. Two commercially available packaging materials (*APP* and *BPE*) were analyzed for their performance in PATP treatments. Samples of materials were used to obtain the bags for food products, in our case pork meat packed under vacuum-conditions.

Pork. The pork used in this study was provided by the Alvino SRL (Romania). The pork meat was brined (10% brine solution containing salt, nitrites and potassium iodide) using Gunter Injektor PI 21 (Germany) and transferred into a GPA 200K equipment (Germany) for tumbling. Then, the pork was sliced (cylindrical shape) and vacuum-packaged with Komet Plus Vac 21 machine (Germany) in individual small bags (7x7cm) from *APP* and *BPE* packaging materials.

PATP experiment. To perform the PATP experiments, the pork packaged meat was PATP treated in a pilot unit built by Resato (Netherlands). The pouches were first preheated into a water bath for a predetermined time at the initial temperature (10 min at 70°C) to reduce the temperature gradients during high pressure treatment. In a next step, the bags were transferred into Teflon cylinders, also equilibrated at the same temperature and containing water at 70°C. These cylinders filled with water and containing the pouches were immediately transferred into the vessels. The vessels were brought at the working temperature with a water bath and the temperature in the vessels was adjusted by an outer heating jacket [12]. The pressurizing liquid was a mixture of glycol and water. The temperature, in the cylinders of the pressure vessels filled with water, was measured by wire thermo-couples feed through the cylinders that allowed monitoring the electrical signal and temperature measurement in the vessels (Fig. 1). The come-up-time for the 600 MPa treatments was set at 30 s and a 60 s time period was selected as an equilibration time, after the pressure build up. All the PATP treatments at constant pressure of 600 MPa were carried out at 70°C for 10 min.

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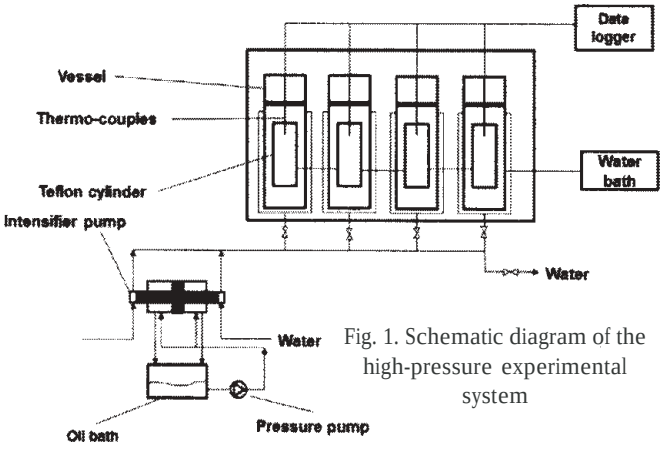

Fig. 1. Schematic diagram of the high-pressure experimental system

Six hundred MPa pressure is considered by many authors as threshold value and also is considered to be economically feasible and microbiologically safe to ensure the pasteurization effect [1, 9, 13]. If we supplement the temperature with high pressure treatment (600MPa), in our case 70°C, the expected effect is comparable with a sterilization effect. PATP treatment was carried out in duplicate for each type of material.

Scanning electron microscopy (SEM). SEM is one of the most widely available tools in surface analysis and it has been used to examine the structure of the packaging materials [14]. The films' surfaces and structures evaluation were carried out using a Quanta 200 microscope (FEI Company, Netherlands) with an operating voltage of 20 kV [15, 16]. The pouches were prepared by cutting 2×2 cm strips from each material. Further the strips were mounted onto the microscope chamber and coated with gold via sputtering. The materials were evaluated at 500x magnifications. A representative number of pictures were taken.

Results and discussions

The pressure/temperature profile

Pressure and temperature were continuously monitored and recorded (1s interval) during the process. The pressure build up was made at a 20 MPa/s rate. The adiabatic compression leads to an increase of the temperature inside the cylinder to almost 90°C. At constant pressure a temperature drop with almost 7°C was noticed due to heat loss through the vessels' walls during pressurization. The heat loss occurs via thermal gradients due to the difference between the lack of compression heating at the steel walls of the vessels and the presence of adiabatic heating of the fluid and of the food which had a matrix with high fat content. Decompression phase suddenly decreased the product temperature below the equilibration temperature at 65°C (fig 2).

Finally, the food product is slowly cooled down to room temperature (25°C).

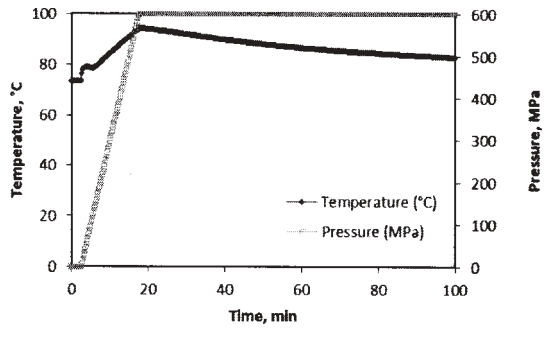

Fig. 2. The pressure/temperature profile

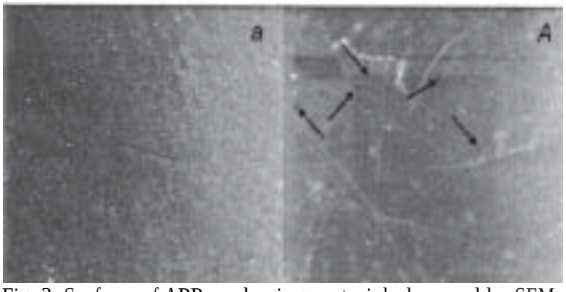

Fig. 3. Surface of APP packaging material observed by SEM: (a) control, (A) treated at 600 MPa, 70°C

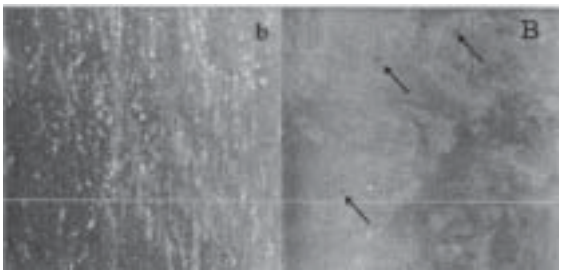

Fig. 4. Surface of BPE packaging material observed by SEM: (b) control, (B) treated at 600 MPa, 70°C

The profile of pressure and temperature differs from the regular isothermal-isobaric processes that are currently applied in many researches and is directly influencing the properties of the product treated by PATP.

SEM analysis

Scanning electron microscopy was used to observe the surface topography and the structure of the *APP* and *BPE* packaging materials. The micrographs of the packaging materials' surfaces and sections before and after PATP are shown in figures 3-6. Figures 3 and 4 illustrate the surface of the *APP* and *BPE* before and after PATP.

The untreated packaging materials (control) show a smooth and homogeneous surface typical of a polymer without pores or cracks, indicated in figures 3a and 4b. The micrographs of treated materials indicate that the PATP generated changes in the polymeric matrix of both *APP* and *BPE* (figs. 3A and 4B). The *APP* material has a non-uniform surface with more scratches and numerous dark spots indicated in graphs by the tip of arrows.

Similar dark spots have been found on the *BPE* indicated in graphs by the tip of arrows (fig. B). The dark spots visualized in figures 3 and 4 images may be holes or deep pits, within the material.

Figures 5 and 6 illustrate the section of the packaging material before and after PATP.

The microstructure was qualitatively studied, aiming to clarify the impact of PATP on the structure of the *APP* and *BPE* packaging materials. Experimental results reported in the literature highlight that the high pressure can compromise the structural integrity of food packaging materials (delamination phenomena) [5]. The cross-sections (indicate by arrows) of both control packaging materials showed multilayer structures, with a compact region at the film-air interface.

The microstructure of the cross-sectional areas of both control packaging materials revealed clearly the individual layers, clearly visible (figs. 5a and 6b). It was obvious from the micrographs that the PATP induces qualitative structural change in all cross-sections of treated packaging materials (figs. 5A and 6B). The cross-sectional area of the treated *APP* material reveals unclear layers and changes in the layer thickness (fig. 5A). These changes could be attributed to absorption of some compounds, such as water

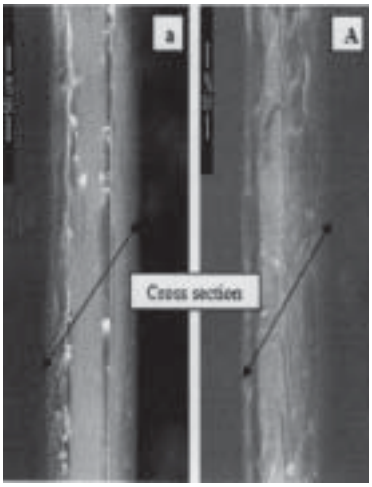

Fig. 5. Section of APP packaging material observed by SEM: (a) control, (A) treated at 600 MPa, 70°C

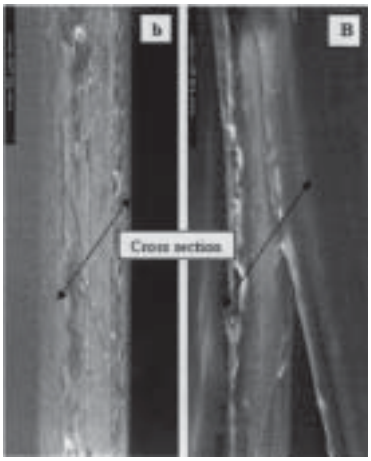

Fig. 6. Section of BPE packaging material observed by SEM: (b) control, (B) treated at 600 MPa, 70°C

from food matrix on the polymer structure. Under the action of water the polymer swells and changes its structure [17]. The micrographs of the treated *BPE* material indicate unclear layers, changes in the layer thickness, increased of the interlayer distance with delamination area (190.87μm in thickness) (fig. 6 B).

Several factors related to the properties of the packaging materials employed to build up the multilayer structures are expected to be responsible for the occurrence of the delamination phenomenon [5]. However, the phenomenon is rather complex and some analyses of the multilayer systems are required before diagnose the transformation that occurs such as mechanical and thermal tests. The bonding conditions at the layer interfaces are also an important issue in terms of cohesive and adhesive properties of lamination adhesives. The unclear layers of the treated films might generate inferior mechanical properties. Although the cause of delamination of multilayer films by high pressure remains to be elucidated, various explanations have been proposed [7]. Some authors showed that the high pressure treatments at pressures higher than 200 MPa caused delamination failure phenomena, such as: PA/PE, Polyester/nylon/aluminum/ Polypropylene and Nylon/EVOH//PE, PE/EVOH/PE [6, 18 - 20]. They postulated that the delamination was due to differences in the elasticity of each layer, the type of glue used the presence of air pockets in the packaging materials and the compressibility between layers [3, 5 - 6, 19 - 20]. Others authors postulated that this defect may arise as a result of the solubilization of gases into the film during pressurization and the subsequent 'escape' of these gases during depressurization [7]. The degree of solubility of these gases in the film layers may influence the degree of delamination [7].

Visual evaluation

During this part of the study, visual examinations were performed on the PATP treated bags at 600 MPa, 70°C and compared with untreated bags. The photos of the packaging materials' surfaces before and after PATP are shown in figures 7 - 10. Figures 7 and 8 depict the surface of the packaging material before and after PATP.

After PATP treatment each bag was examined for visual potential defects, such as: delamination, opaque areas, pits, bubbles or other pressure-temperature induced defects. The treated bags were showing after the treatment visible changes, such as: opaque areas and delaminations. These changes are presented in figures 9 and 10.

After PATP, *APP* material showed small white opaque areas (fig. 9). In case of the *BPE* material, larger and far more prevalent opaque areas and delaminations were presented, compared to the *APP* material (fig. 10).

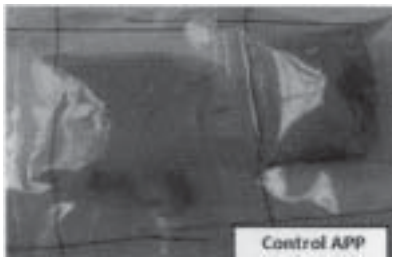

Fig. 7. Photos on untreated APP packaging material

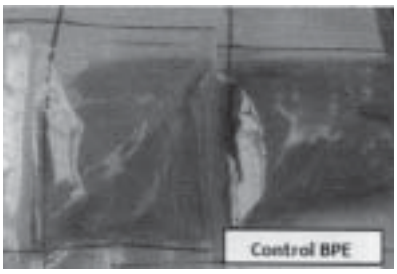

Fig. 8. Photos on untreated BPE packaging material

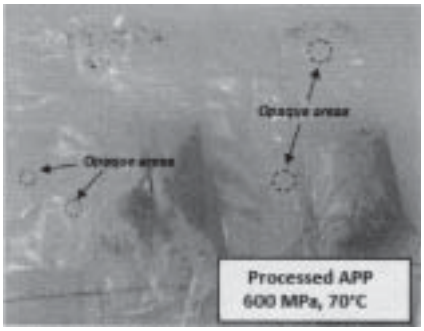

Fig. 9. Opaque areas on APP material after 600 MPa, 70°C

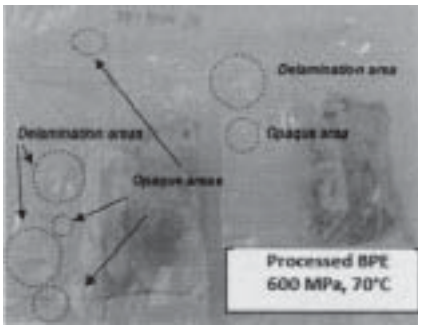

Fig. 10. Opaque areas and delaminations on BPE material, after 600 MPa, 70°C

The opaque and delaminations areas could be a consequence of incompatibility between layers in multilayer packaging material [21] and/or a rapid and explosive decompression of solubilized gases (nitrogen and oxygen) within material [7, 15].

Film thickness's evaluation

The thickness was measured by a Quanta 200 microscope (FEI Company, Netherlands). Measurements were taken at five different locations on the material sample and a mean thickness was calculated [22]. The materials thicknesses before and after PATP are reported in table 1.

Table 1
THE PACKAGING MATERIALS' THICKNESS (μm)

Packaging material	Control	600 MPa 70°C	Deffect
APP	55.95±0.00	60.90±0.29	-
BPE	80.00±0.22	70.52±0.15	190.87

It is known that the high pressure treatment can accelerate the mass transfer through, from or into packaging materials [8].

From the table 1 data it can be observed that the *APP* sthickness increased from 55.95±0.00μm to 60.90 ±0.29μm, similar to other multilayer complex material such as PA/EVOH/PA/PE (PA - polyamide, EVOH - ethylene vinyl alcohol, PE - polyethylene) [4]. In case of *BPE* material, a thickness' decrease from 80±0.22μm to 70.52±0.15μm can be observed, similar to other multilayer material (combination of biaxially oriented polyamide with coextruded barrier film of the structure PE/EVOH/PE) [4]. In addition, the processed *BPE* material indicates a thickness increase in a certain area to 190.87 μm (fig. 5B).

In case of thickness's decrease it should be taken into account a mass transfer (migration) phenomenon from the packaging material (non-volatile migrants: monomers, additives: plasticizers, light stabilizers or other volatiles compounds) to the food matrix. The migration can increase with the increase of the fat content because most of the plastic material constituents are lipophilic rather than hydrophilic [21]. In addition, with the absence of experimental data for film's solubility at experimental PATP parameters, to support these statement further studies are needed, such as: GC (gas chromatography) and GC-MS (GC-mass spectrometry). In case of thickness's increase it should be taken into account an interlayer failure phenomena, mainly for *BPE* packaging material (fig. 4 A and table 1) or it could be attributed to absorption of some compounds from food matrix on the polymer structure, mainly for *APP* packaging material.

Conclusions

In general, the results showed that the combined pressure-thermal treatment equivalent to a sterilisation effect in food products alters the tested packaging materials. Opaque areas and delaminations were a general consequence of the pressure-assisted thermal processing of tested packaging materials at 600MPa combined with 70°C. Even if the *APP* material was found to be better than *BPE* material, the tested materials don't represents options for packaging foods treated PATP, because the changes in the packaging materials, even the small opaque areas noticed after the treatment, could compromise the integrity, the stability and the aesthetic qualities of the packaging materials.

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